PREPARATION AND CHARACTERIZATION OF ALUMINA- STRONTIUM ALUMINATE BASED COMPOSITE

A THESIS SUBMITTED IN THE PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF
BACHELOR OF TECHNOLOGY

By

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CONTENTS

NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA

CERTIFICATE

This is to certify that the thesis entitled, **"Preparation and characterization of strontium aluminate based composite"** is the bonafide work of Miss **Manali Madhuchhanda (Roll no- 111CR0480)** in partial fulfilments for the requirements for the award of ***Bachelor of Technology*** degree in ***Ceramic Engineering*** at National Institute of Technology, Rourkela is an authentic work carried out by her under my supervision and guidance. Certified further, that to the best of my knowledge the work reported does not form part of any other thesis or dissertation on the basis of which a degree or award was conferred on an earlier occasion on this or any other candidate.

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ABSTRACT

Alumina-Strontium aluminate composites were prepared by mixing of Al_2O_3 and $Sr(NO_3)_2$ and calcination of powder at 1100^oC. The calcined powder was compacted and sintered at 1600^oC . The calcined powder XRD reveal that about 7% SrO. Al_2O_3 has formed in-situ in Al_2O_3. The sintered sample had a poor relative density (75% of theoretical). The sintered samples were characterised by apparent porosity, bulk density, hardness, strength and toughness. The hardness of sintered sample was 2.49 GPa, biaxial flexural strength was 37.3 MPa and Indentation Strength in Bending(ISB) fracture toughness of 1.7 $MPam^{1/2}$. SEM microstructure of the sintered samples exhibits a porous microstructure. The strontium aluminate particles were seen as bright particles in phase contrast mode. The crack path was also viewed in SEM. It was noted that the cracks either were branched on interacting with strontium aluminate or stopped. This feature indicates the possibility of producing a tough sample if proper density and homogeneous distribution of Strontium aluminate could be achieved.

LIST OF FIGURES

LIST OF TABLES

1. INTRODUCTION

INTRODUCTION

Alumina is among the important ceramic materials that have versatile utility. The high hardness, excellent corrosion and wear resistance of alumina have been utilised in many demanding applications such as seals, wear liners, grinding media, cutting tool inserts, etc. However, the low fracture toughness of alumina often leads to catastrophic failures leading to premature failure of these materials. Many attempts have been made to improve the resistance to crack propagation or fracture toughness. Some of the examples are (a) the addition of ductile particles in alumina matrix (Cu in Al_2O_3 matrix) [1], (b) reinforcement with stainless steel fibres in refractories [2], and (c) incorporation of secondary phases like TZP, TiC etc. in Alumina [3]. All the above categories are part of dispersion strengthening ceramics. Al_2O_3 can also be reinforced by incorporating calcium aluminate [4,5], yttrium aluminate [6], aluminium titanate [7], lanthanum aluminate [7], etc. In a similar manner, Strontium aluminate [7] has also been introduced in the Al_2O_3 matrix to improve its mechanical properties particularly strength and toughness. It is claimed that in most of the above aluminates, the elongated grains give rise to improved properties through crack deflection and crack bridging mechanisms [4,5,7].

The extent of strengthening and toughening depends on the volume fraction of the second phase as well as on the nature of secondary phases (i.e. aluminates), grain size and shape [4,5,7]. Some of these aluminates have been pre-processed through mixing and calcination of alumina and the metal oxide (CeO_2, La_2O_3, SrO) and then mixed with alumina in different proportions to fabricate the composites [6]. In other instances, the metal oxide is mixed with matrix and sintered at a high temperature whereby the formation of aluminates and densification of the composites simultaneously takes place [8].

In the light of above discussion, the present study was undertaken to observe the effect of strontium aluminate incorporation on the densification and properties of Al_2O_3 composites.

2. LITERATURE REVIEW

LITERATURE REVIEW:-

The literature review has revealed that the low fracture toughness of alumina can be improved by the incorporation of a second phase with a high aspect ratio like fibres, whiskers, and platelets. Chen et al. [9] has reported that an in-situ formation of highly anisotropic secondary phase in the alumina matrix could result in high toughness of alumina based composites. Chen et al. reported that Al_2O_3 is compatible with many layer type aluminate compounds such as La_2O_5, SrO, CaO, etc. The incorporation of these oxides can increase the toughness of alumina through the formation of secondary aluminate phase.

Kern et al.[3] reported that the mechanical properties of alumina can be also improved by the incorporation of 20-30 vol% of 3Y-TZP in Al_2O_3. These Alumina toughened zirconia (ATZ) were observed to have high toughness provided 3Y-TZP existed as a combination of cubic and transformable tetragonal phase Kern et al. reported strength values up to 2 GPa and a toughness of 4.5-5 MPa $m^{1/2}$ in these composites.

Sanchez-Herencia et al. [4] has studied the fracture behaviour of Alumina-Calcium Hexa – Aluminate ($CaAl_{12}O_{19}$) based composites. It was observed that the critical fracture energy and, therefore, the toughness of alumina materials depended upon the grain size, shape and microstructure. It was observed that up to a critical grain size micro crack generation was the dominant toughening mechanism. However at larger grain sizes, crack branching was the primary toughening mechanism. Microcrack toughening resulted from the thermal expansion mismatch between alumina matrix and calcium hexa-aluminate that had lower thermal expansion.

Shuai et al. [10] also studied the microstructure and the change in mechanical properties by the incorporation of Calcium hexa-aluminate (CA_6). CA_6 developed into a plate-shaped morphology and a layered structure.The phase was also stable to the peritectic point of hexa-aluminate (1875^oC). The presence of elongated CA_6 grains toughened the composite via crack-deflection and crack bridging mechanisms. Due to better toughness, it could prevent the crack propagation and improve the spalling resistance.

Vishista et al. [5] has shown that the incorporation of in situ platelets of calcium hexa-aluminate during processing resulted in enhanced mechanical properties of ceramic materials. The mechanical properties of alumina composites depended on grain size, shape, and grain size distribution. It was shown that incorporation of in-situ secondary phase during densification

led to the development of randomly dispersed phases with plate-like elongated structure. Due to the plate-like morphologies the elongated grains acted as bridging sites in the wake of the crack and thus increased the toughness and mechanical properties. The improved toughness was principally due to crack stabilization from the grain bridging. The authors further showed that the sol-gel process was one of the most effective methods to synthesize such composites.

Theeranee et al.[6] has studied the characteristics and mechanical properties of alumina matrix based composites containing zirconia and strontium oxide. The ZrO_2 addition to alumina acted as grain growth inhibitor of alumina and resulted in finer grain size of alumina. The combination of fine grain size of alumina and transformation toughening effect induced by ZrO_2 particles inhibited crack propagation of the matrix. The use of SrO resulted in elongated grains of strontium aluminate enhancing toughness due to crack deflection. The authors reported that for the incorporation of 15 vol% of SrO the relative density was found to be 0.59 gm/cc, hardness of 1.03 GPa and toughness value of 1.54 MPam1/2.

Vishista et al. [7] incorporated SrO in alumina sol and studied the improvement in the mechanical properties of the sintered composites. Hardness, fracture toughness, diametrical tensile strength, bi-flexural strength had been studied for composites with various concentrations of strontium oxide addition. The microstructure having in-situ Strontium aluminate leads to high mechanical properties due to the formation of anisotropic elongated grains. The size of the plate-like grains in the sintered bodies was found to have an effect on the extent of toughness increment.

Many studies have been reported showing improvement in strength in alumina matrix due to the presence of hexa-aluminate phases. Cutler et al.[11] showed that incorporation of strontia as $SrZrO_3$ into Ce-TZP/Al_2O_3 composites provided higher flexural strength and fracture toughness. Maschio et al. also found similar results [12] and reported excellent toughness in Al_2O_3-Cr_2O_3/ZrO_2 composites with the addition of small amount of SrO.

3. AIM OF WORK AND PLAN OF WORK

3.1 AIM OF THE WORK:-

In the light of above discussion, the present study was undertaken to observe the effect of strontium aluminate incorporation on the densification and properties of Al_2O_3 composites.

The study involved the following:-

- To prepare strontium aluminate-The strontium aluminate was developed in-situ through the mixing of Sr $(NO_3)_2$ and Al_2O_3 powder followed by calcination at 1100^oC.
- Phase analysis of the calcined powder.
- Compaction and sintering of the calcined powder at 1600^o C.
- Phase analysis of the sintered powder.
- Study of bulk density, apparent porosity, biaxial flexural strength, hardness and fracture toughness of sintered samples.
- SEM study to observe the distribution of Al_2O_3 and strontium aluminate phase.
- Indentation of some of the samples in a Vickers Hardness Tester and tracking of the crack path of the indented samples to study the effect of strontium aluminate phase on crack propagation path.

3.2. PLAN OF WORK-

The scheme of work for this study is as follows:-

- Processing of Al_2O_3- Strontium Aluminate composite powder
- DSC/TG of dried powder
- Calcination of composite powder
- Phase analysis of calcined powder
- Dilatometry of powder compact
- Compaction and sintering of pellets
- Phase analysis of sintered pellets
- Property Evaluation
    - Linear shrinkage
    - Apparent porosity
    - Bulk density
    - Hardness
    - Bi-flexural strength measurement
    - Fracture toughness measurement
    - Microstructure of the sintered and indented composite

4. EXPERIMENTAL PROCEDURE

EXPERIMENTAL PROCEDURE –

4.1. RAW MATERIALS:

In this work, the starting raw materials were calcined alumina (source) and SrO (source). SrO was dissolved in HNO_3 and the prepared $Sr(NO_3)_2$ was mixed with Al_2O_3 so as to yield 15 mol % SrO on calcination.

For the formation of $SrO.Al_2O_3$, first we need to prepare the powders of SrO and calcined Alumina.

1. Processing of as received alumina powder
2. Estimation of Sr $(NO_3)_2$ stock solution
3. Preparation of Al_2O_3- Sr $(NO_3)_2$ mixed powder
4. The batch calculation for 25 gm of the batch is done to estimate the amount of SrO and Al_2O_3 required for each composition.

4.1.1. PROCESSING OF AS RECEIVED ALUMINA POWDER-

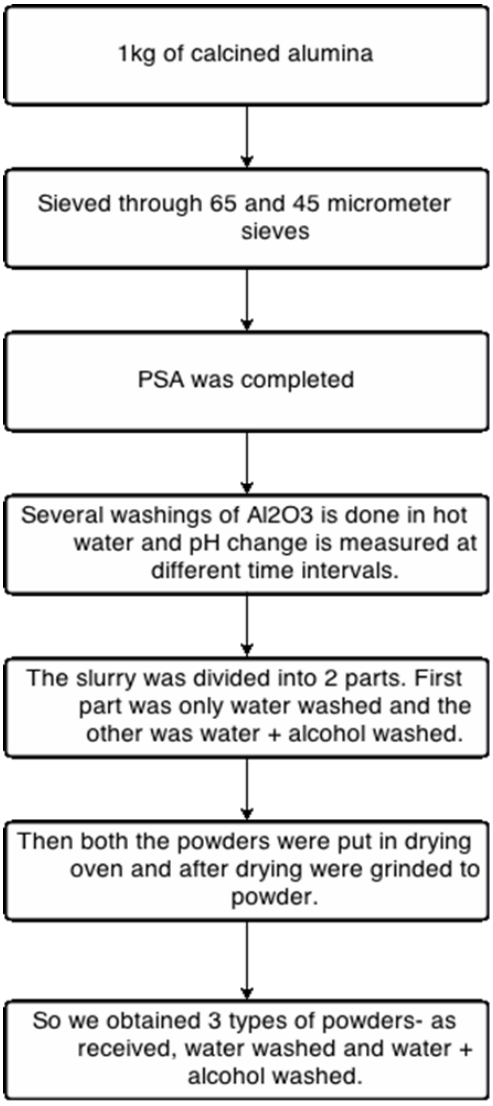

FIGURE 1- FLOW CHART FOR THE PROCESSING OF WATER+ALCOHOL WASHED ALUMINA

The particle size analysis for water + alcohol washed alumina is shown in fig 2. Since the agglomeration formation rate is lower than the other two types of alumina, water + alcohol washed alumina is used for the preparation of this composite.

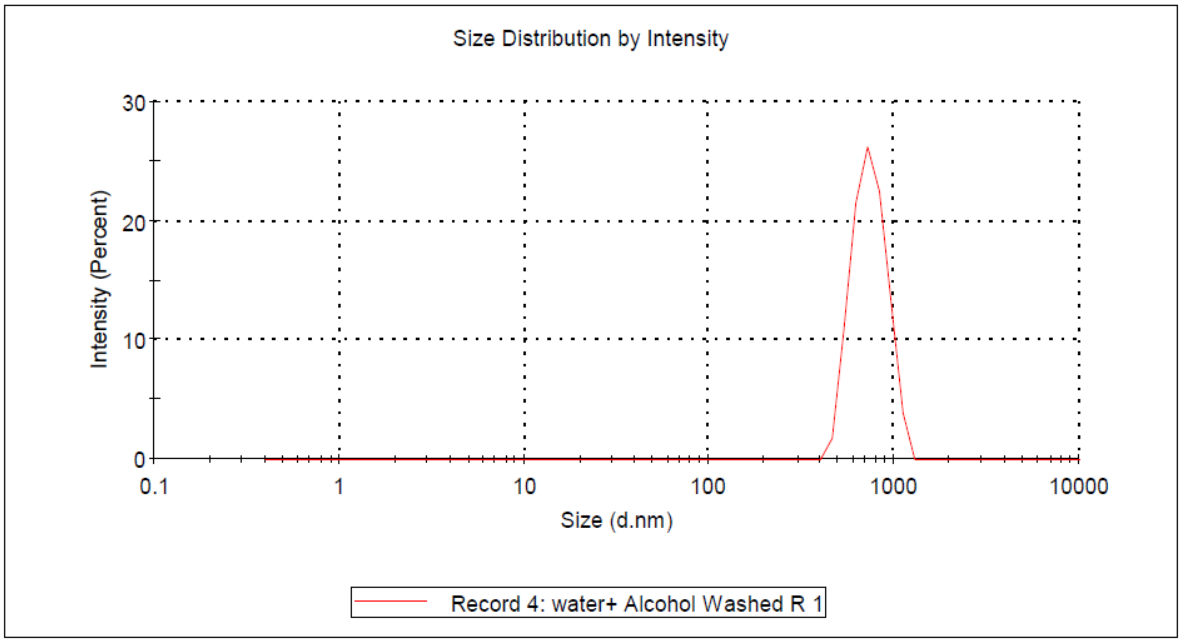

FIGURE 2- PARTICLE SIZE ANALYSIS FOR WATER+ALCOHOL WASHED ALUMINA

4.1.2. Estimation of SrO in Sr $(NO_3)_2$ solution:-

- $Sr(NO_3)_2$ is dissolved in water and stirred until dissolved.
- The impurities are filtered, and the empty crucible is pre-weighted.
- Ammonia is added to 2 ml of the sample until it smells of ammonia.
- Then the sample is dried in an oven at 80^oC and then kept in a furnace at 1000^oC in 2 hour.
- Then the sample is cooled and weighed with lid.
- So, the weight of SrO is found out to be 0.5858 gm in 2 ml of solution.

4.2.BATCH CALCULATION –

For 25 gm batch,

Molecular weight of Al_2O_3 = 102

Molecular weight of SrO = 103.6

1 mole of SrO = 103.6 gm

0.15 mole of SrO = 103.6 * 0.15 = 15.54 gm

1 mole of Al_2O_3 =102 gm

0.85 mole of Al_2O_3 = 102*0.85 gm = 86.7 gm

1 mole of mixture= 86.7 + 15.54 gm =102.24 gm

102.24 gm of mixture = 1 mole

25 gm of mixture = 25 /102.24= 0.2445 mole

In 0.2445 mole,

Amount of SrO = 103.6 *0.0366 = 3.791 gm

Amount of Al_2O_3 =0.2078 *102 = 21.198 gm

To get 15 gm of Al_2O_3 ,

Amount of $Sr(NO_3)_2$ = 5.46 gm

Now, the solubility as calculated above was found out to be 60 gm in 100 ml.

So, 9.1 ml of $Sr(NO_3)_2$ is required for the preparation of 15 gm of Al_2O_3 .

4.3. CALCINATION, POWDER COMPACTION-

The required mixture of Al_2O_3 and SrO are taken and stirred in a magnetic mixer till a homogeneous mixture is obtained. Then the mixture is taken in a Petri dish and is dried. Then the mixture is ground into fine powder by using agate mortar and then calcined at around 1100^OC for 2 hours. XRD analysis was done on the calcined powder, and it showed the presence of $SrO.Al_2O_3$. 3% PVA was added to the calcined powder that was obtained and fired at 1600^OC. Pellets were then made out of this fired powder for further characterization.

4.4. CHARACTERIZATION-

4.4.1. DSC/TG – DSC/TG (STA449C- NETZSCH Germany) is used to determine the phase transitions and chemical reactions by measuring the difference in the amount of heat required to increase the temperature of a sample and reference and are recorded as a function of temperature. We can find out the compounds decomposing and the phase transitions occurring during the reaction. The decomposition and phase formation reactions can be studied by this technique.

4.4.2. X-ray Diffraction- It is a versatile, non-destructive technique that reveals detailed information about the crystallographic phases of the manufactured materials. XRD is a powerful tool used to detect the presence of phases in the material. The main principle behind XRD is Bragg's law that states

$$n\lambda = 2d\sin\theta \quad (1)$$

Where, d = spacing between diffracting planes,

θ = incident angle,

n = any integer

λ = wavelength of the beam

As the study of the various phases and their analysis is the main objective of this project work, XRD analysis was performed. The XRD scan was done by Philips' X-ray diffractometer with Nickel filtered Cu Kα radiation (1.5406A^o). The diffraction pattern was between 15°-60^o at a scanning speed of 2^o/min. From the XRD plot, peaks corresponding to the different phases were analysed and also the composition of important phases was studied.

4.4.3. DILATOMETRY-

(DIL402C, NETZSCH Germany) The dimensional changes of the sample versus temperature or time are measured using pushrod dilatometry while the sample undergoes a controlled temperature program.

4.4.4. LINEAR SHRINKAGE – Linear shrinkage is defined as the reduction in the dimension of the sample due to firing of the sample.

$$\text{Shrinkage (\%)} = [(L_{\text{before firing}} - L_{\text{after firing}})/L_{\text{before firing}}] \times 100 \quad (2)$$

4.4.5. BULK DENSITY-

The bulk densities of the sintered pellets were determined by Archimedes principle using the mass in air and the mass when immersed in water. For Dry Weight, the sample is weighed in the air.

The weighed sample was kept in a beaker filled with kerosene and evacuated for one and half hour so that all the pores present in it were filled with kerosene and when air bubbles stopped the vacuum pump was turned off. The suspended weight and soaked weight of the samples were also taken. Weight is taken by wiping off the excess kerosene present on its surface using a wet cloth. Once the dry weight, soaked weight and suspended weight were measured, bulk density and apparent porosity were calculated by the formulas:

B.D. = Weight in air / (soaked weight – suspended weight) x Liquid density

4.4.6. APPARENT POROSITY- The apparent porosity is a measure of the volume of the volume of the open pores into which a liquid can penetrate, as a percentage of the total volume. A low apparent porosity is desirable since it would prevent easy penetration. A large number of small pores have an important influence on the mechanical properties. However, a measure of the true porosity, which also takes into account the volume of closed pores, gives a reasonable idea of the texture of the material as well as sintering characteristics.

The Apparent Porosity is measured from the dry weight, soaked weight and suspended weight. It can be expressed as:

$$\text{A.P. (\%)} = [(\text{Soaked weight} - \text{Weight in air}) / (\text{soaked weight} - \text{suspended weight})] \times 100 \quad (3)$$

4.4.7. HARDNESS- The property of a material that enables it to resist plastic deformation, usually by penetration is termed as hardness. Hardness also refers to resistance to bending, scratching, abrasion or cutting.

MEASUREMENT OF HARDNESS –

Hardness is measured by Vickers's method. In Vickers's method, the test sample is indented with a diamond indenter in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 kgf. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and the average diagonal length is calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of the indentation.

The formula for the calculation of hardness is given as follows:-

$$\text{HV} = 1.854\frac{F}{d^2} \text{ (approximately) } [13] \qquad (4)$$

Where HV= Vickers's hardness

F = Load applied

D= arithmetic mean of the two diagonals, d1 and d2 in mm

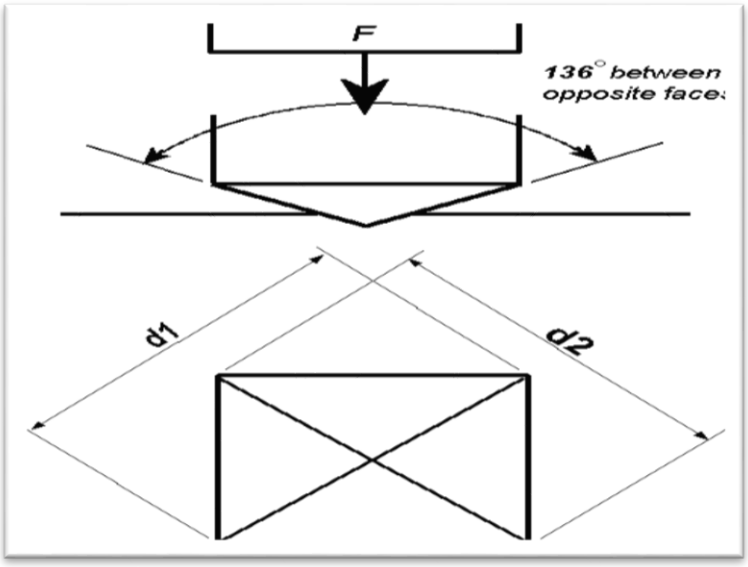

FIGURE 3:- INDENTATION OF SAMPLE FOR VICKER'S HARDNESS TEST [14]

4.4.8. BIFLEXURAL TENSILE STRENGTH MEASUREMENT-

For this measurement, the pellets were kept diametrically under the load. Biaxial Flexural Strength = 2*Force/ (3.14 * Diameter * Thickness)

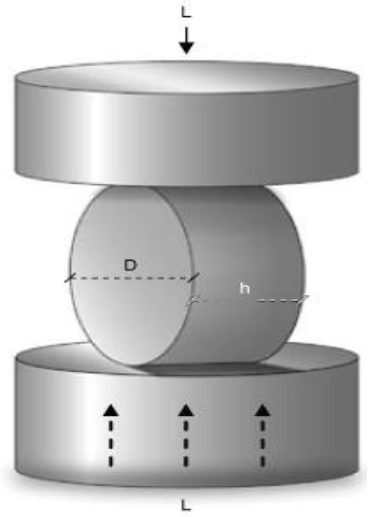

FIG 4:- DIAGRAM OF DIAMETRICAL TENSILE STRENGTH TEST SET UP [15]

Bi-flexural strength was measured by the Universal Testing Machine (HKIOS, Tinius Olsen UK). The test procedure involves placing the test specimen along the diameter as shown in fig 3. in the testing machine and slowly compressing it until it fractures.The test specimen alignment in the testing machine is critical because if the specimen is misaligned, either at an angle or offset to one side, the machine will exert a bending force on the specimen. This test can determine the strength and the fracture toughness if the sample is indented.

4.4.9. FRACTURE TOUGHNESS MEASUREMENT – Fracture toughness is measured by Indentation Strength in Bending (ISB). For this, circular disc specimens (dia 25 mm diameter and 2 mm thickness were indented at 20 kgf with dwelling time 20 sec at the centre of the sample in an LECO Vickers's Indenter Hardness tester. The indented sample was broken by ring on ring test method at crosshead speed of 0.5 mm/min, and the toughness value was calculated from this formula:-

$$K_{1C} = \tilde{\eta} \left(\frac{E}{H}\right)^{\frac{1}{8}} * (\sigma * P^{1/3})^{3/4} \quad (5)$$

Where E= elastic modulus (MPa), P= load applied (Newton), σ = fracture strength of the sample.

The fracture strength can be calculated from the following equation:-

$$\sigma = \frac{3F}{2\pi h^2}\left[(1-v)\frac{D_S^2 - D_L^2}{2D^2} + (1+v) ln \frac{D_S}{D_L}\right. \quad (6)$$

Where σ = bi flexural strength (GPa), F= load given till material fails (Newtons), h= sample thickness (mm), v= Poisson's ratio , D_S = Support ring diameter (mm), D_L = Upper ring diameter (mm), D = Sample diameter (mm).

4.4.10. MICROSTRUCTURAL OBSERVATION BY FESEM –

(Nova NanoSem 450, made by FEI) It is used to visualize and form images of very small topographical details like cracks, indents, pores on the surface of fractured objects. The basic principle is that the electrons that are emitted from a field emission source and accelerated in a high electrical field gradient bombards the object. As a result, secondary electrons are emitted from each spot on the object. A detector catches the secondary electrons and produces an electronic signal. This signal is amplified and transformed to a video scan-image that can be seen on a monitor. The fractured and indented samples were observed in FESEM.

5. RESULTS AND DISCUSSIONS

RESULTS AND DISCUSSION:-

5.1. DSC/ TG-

Fig (5) shows the DSC/TG plot of dried Al_2O_3- Sr $(NO_3)_2$ mixture. The DSC plot shows an endothermic reaction is occurring at 600^O C and the reaction involves nearly 7% weight loss. Thus, this reaction corresponds to the decomposition of Sr $(NO_3)_2$ to SrO. Between 900^OC - 1100^OC, a broad exothermic peak is observed. There is no weight loss corresponding to the exothermic peak. Hence, the broad exothermic peak corresponds to the phase formation reaction between SrO and Al_2O_3 leading to the formation of Strontium aluminate.

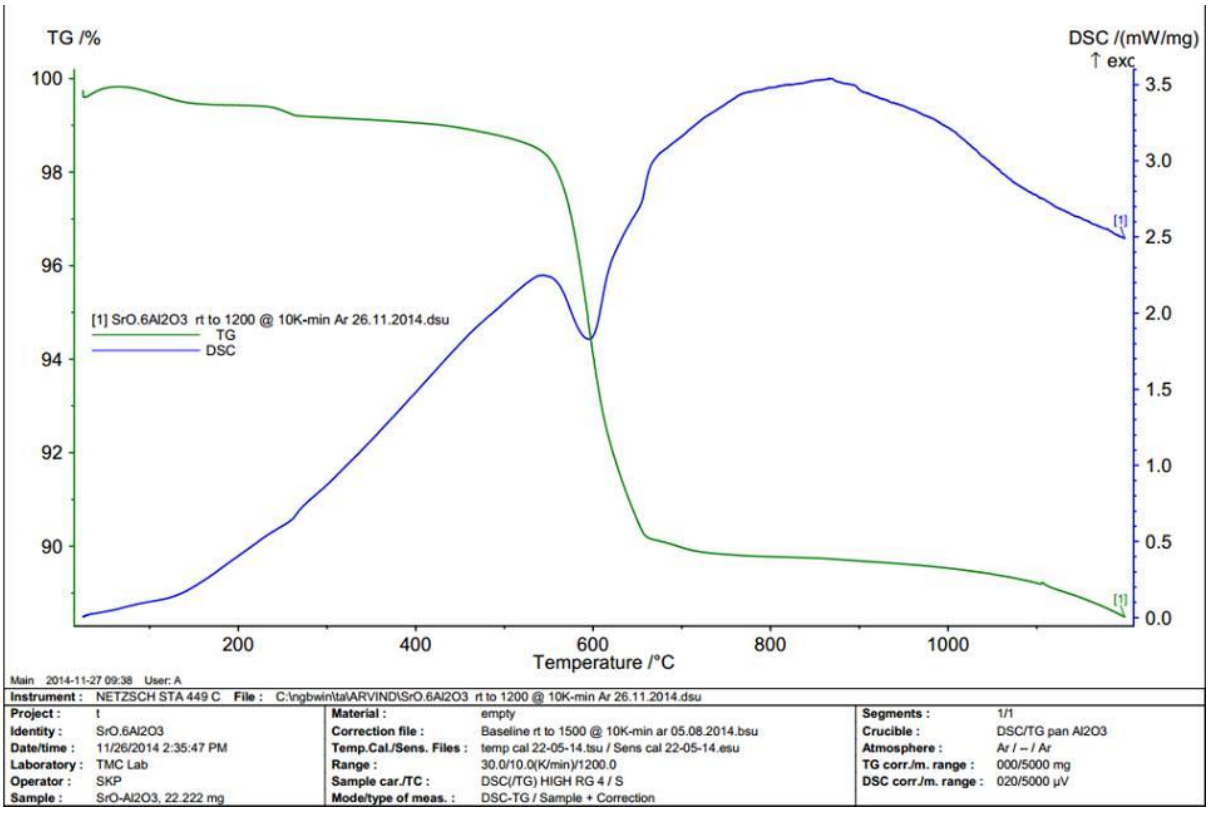

FIGURE 5:- DSC/TG OF THE ALUMINA-STRONTIUM NITRATE COMPOSITE POWDER

5.2. PHASE ANALYSIS-

Fig (6) shows the XRD pattern of calcined powder. The pattern shows that the major peaks correspond to corundum (d-spacing =2.54) and SrO. Al_2O_3 (d-spacing =3.14). Thus, in the calcined powder both Al_2O_3 and strontium aluminate co-exist. The relative volume fraction of these two phases were calculated from their intensity values, and the volume fraction was 0.93, 0.07 respectively.

For sintered sample, the phases identified are that of alumina, corundum and strontium aluminate.

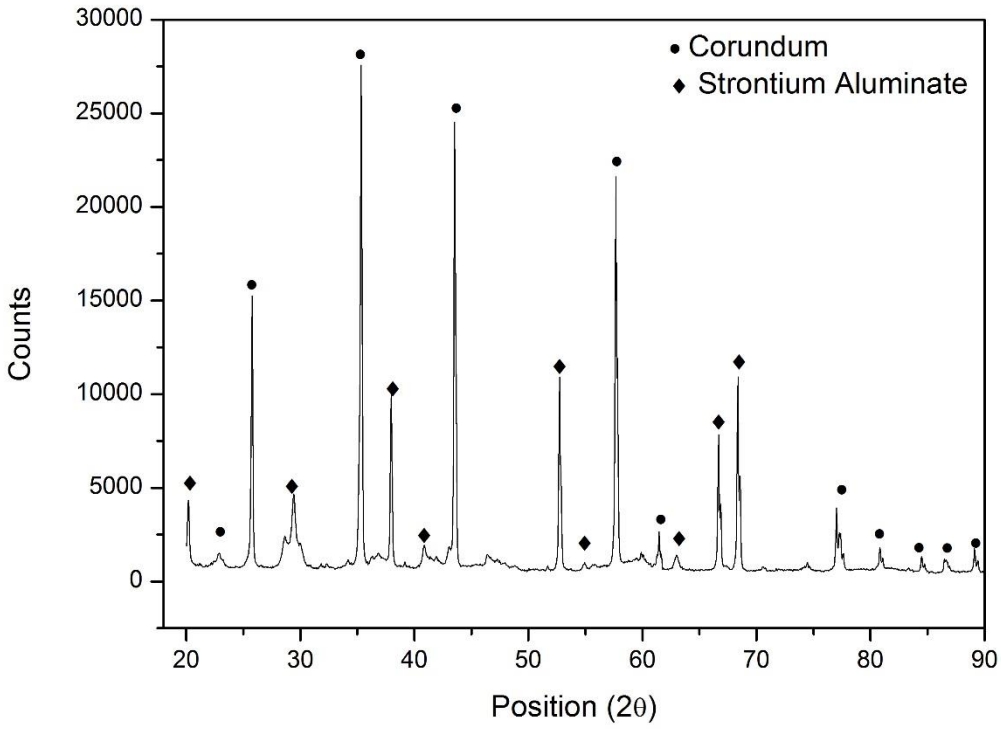

FIGURE 6:- XRD OF CALCINED SAMPLE OF THE COMPOSITE

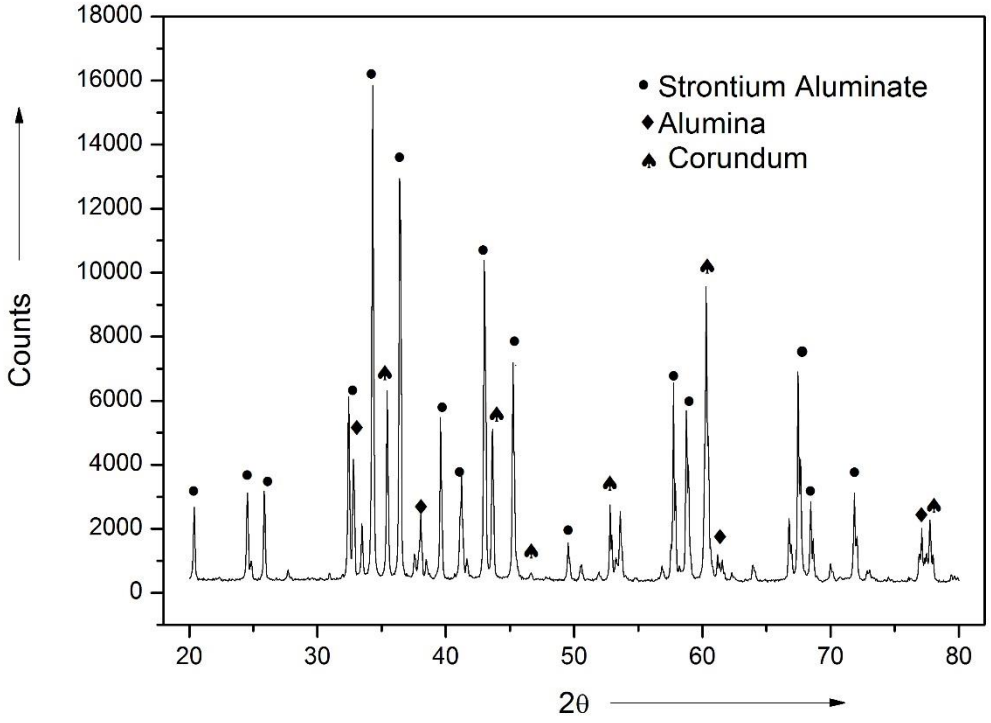

FIGURE 7:- XRD OF SINTERED SAMPLE OF THE COMPOSITE

5.3. DILATOMETRY

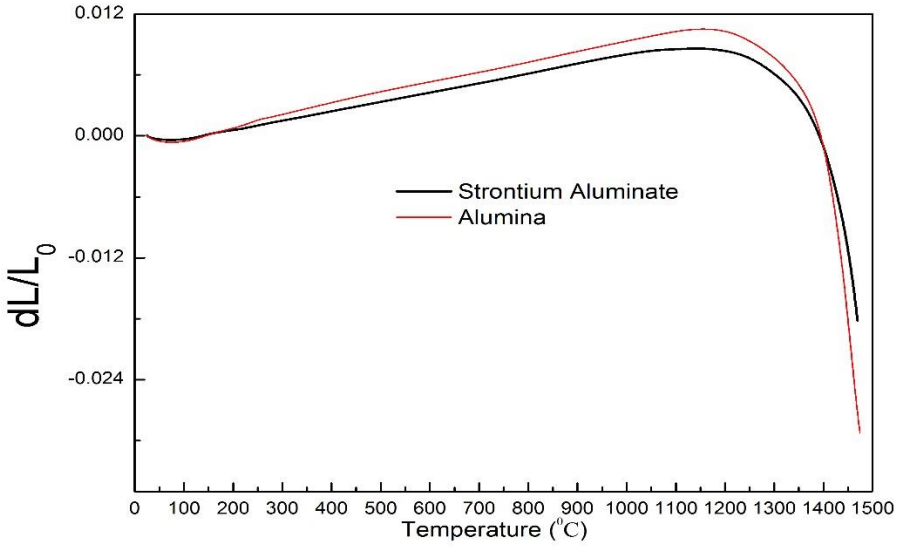

FIGURE 8: MEASUREMENT OF SHRINKAGE BY DILATOMETRY

The dilatometer plot shows that the densification of alumina starts at 1200^{o}C and the total shrinkage is 2.4%. The alumina-strontium aluminate composite in the other hand starts densifying at about 1250^{o}C and only 1.8% shrinkage is observed till 1500^{o}C. Thus, strontium aluminate retards the densification of alumina. However, in either of the two cases, densification is incomplete which implies that the sintered samples are porous.

5.4.LINEAR SHRINKAGE –

TABLE (1)- LINEAR SHRINKAGE OF COMPOSITE AND ALUMINA

SAMPLE	LINEAR SHRINKAGE (%)
Alumina- 7 vol% strontium aluminate composite	10.21
Pure alumina	19.2

From the above table, it can be concluded that the linear shrinkage is increasing as the pellet size is increasing. The linear shrinkage of SrO.Al2O_3 is less than that of Al_2O_3 which indicates that the strontium aluminate based composite has more porosity than alumina pellet.

5.5. BULK DENSITY AND APPARENT POROSITY-

According to the rule of mixtures,

The theoretical density of the composite = density of Al_2O_3 * volume fraction of Al_2O_3 + density of SrO.Al_2O_3 * volume fraction of that phase.

Theoretical density = 3.95 *0.909 + 3.56 * 0.07

= 3.93 gm/ cc

TABLE (2)- MEASUREMENT OF BULK DENSITY AND APPARENT POROSITY

SAMPLE	BULK DENSITY	APPARENT POROSITY (%)	RELATIVE DENSITY (%)
Alumina- 7 vol% strontium aluminate composite	2.447	29.59	62.26
Al_2O_3	3.39	13.11	85.82

Thus, the introduction of SrO. Al_2O_3 in Al_2O_3 retards the densification of Al_2O_3.

5.6. HARDNESS-For the pellets of 12.5 mm diameter, different loads were applied, and hardness values are calculated from the load vs. area^2 plot. Hardness is calculated from the following formula:-

$$HV = 1.854 \frac{F}{d^2} \quad [12] \qquad (7)$$

The slope of the plot gives the $\frac{F}{d^2}$ value.

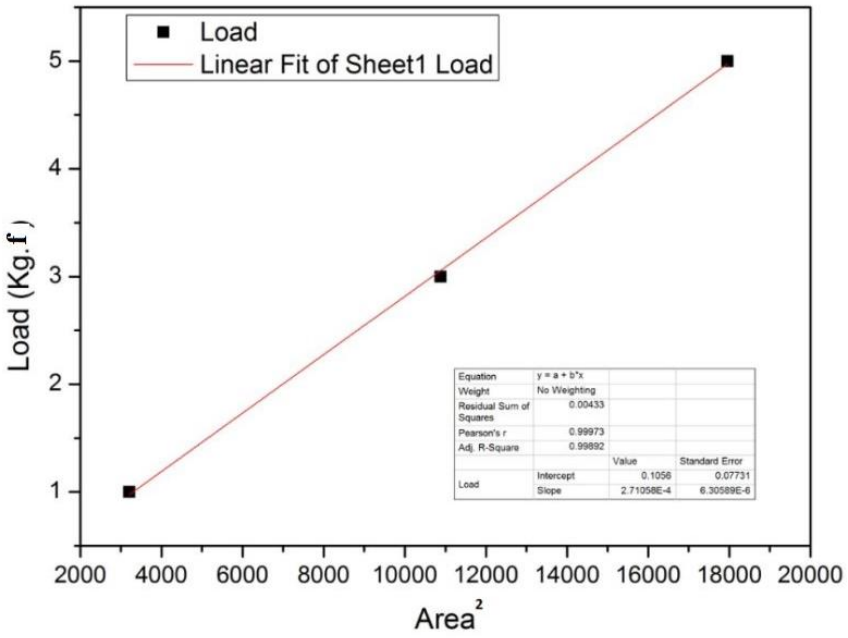

FIGURE 9 :- MEASUREMENT OF HARDNESS BY VICKER'S HARDNESS TEST

For 25 mm pellets, there were two samples, $SrO.Al_2O_3$ and that of Al_2O_3, the hardness values are:-

TABLE (3) – HARDNESS VALUES FOR ALUMINA AND COMPOSITE

SAMPLE	HARDNESS (GPa)
Alumina- 7 vol% strontium aluminate composite	2.492
Al_2O_3	15.895

From the hardness value of the composite, it can be seen that the hardness is low. The low value of hardness is due to more porosity in the sample. More dense is the body; more will be the hardness value.

5.7. BIFLEXURAL STRENGTH-

Bi flexural strength is measured by using the following formula:-

$$\sigma = \frac{3F}{2\pi h^2}\left[(1-v)\frac{D_S^2 - D_L^2}{2D^2} + (1+v) ln \frac{D_S}{D_L} \right] \quad (8)$$

Where σ = bi flexural strength

F= load given till material fails (Newtons), h= sample thickness (mm), v= Poisson's ratio , D_S = Support ring diameter (mm), D_L = Upper ring diameter (mm), D = Sample diameter (mm).

The bi flexural strength was found to be 37.3 MPa.

5.8. FRACTURE TOUGHNESS- Fracture toughness value can also be calculated from this test. The fracture toughness can be calculated from this formula:-

$$K_{1C} = \tilde{\eta} \left(\frac{E}{H}\right)^{\frac{1}{8}} * (\sigma * P^{1/3})^{3/4} \quad (9)$$

E = Elastic modulus (GPa), H= Hardness (GPa)

The measured fracture toughness was 1.71 MPa $\sqrt{m}$.

- The low value of strength and toughness is due to the presence of porosity in the sintered specimen. Thus, for increasing the strength and toughness, the body has to be dense with low porosity.

DIAMETRAL STRENGTH- Diametral strength is measured by keeping the sample diametrically on the UTS. Diametral strength is calculated according to the formula stated earlier. It is found out to be 25.797 MPa.

5.9. MICROSTRUCTURE OF SINTERED SPECIMEN- The microstructure of the sintered and indented samples of alumina-strontium aluminate is shown in fig. (10 -16).The images were taken in Back Scattered Electron Mode. The samples were sputter coated with platinum to minimise charging effect. Fig 10 and 11 show the indentation image on alumina sample. Cracks had originated from the corner of the diagonals, and from the phase contrast it is seen that the microstructure has only one type of grains. Fig. 12 shows the microstructure of Al_2O_3 - Al_2O_3-SrO composite. The microstructure is porous. Two phases can be seen that of SrO. Al_2O_3 and Al_2O_3. The bright region is that of SrO. Al_2O_3 and the dark region is that of Al_2O_3.

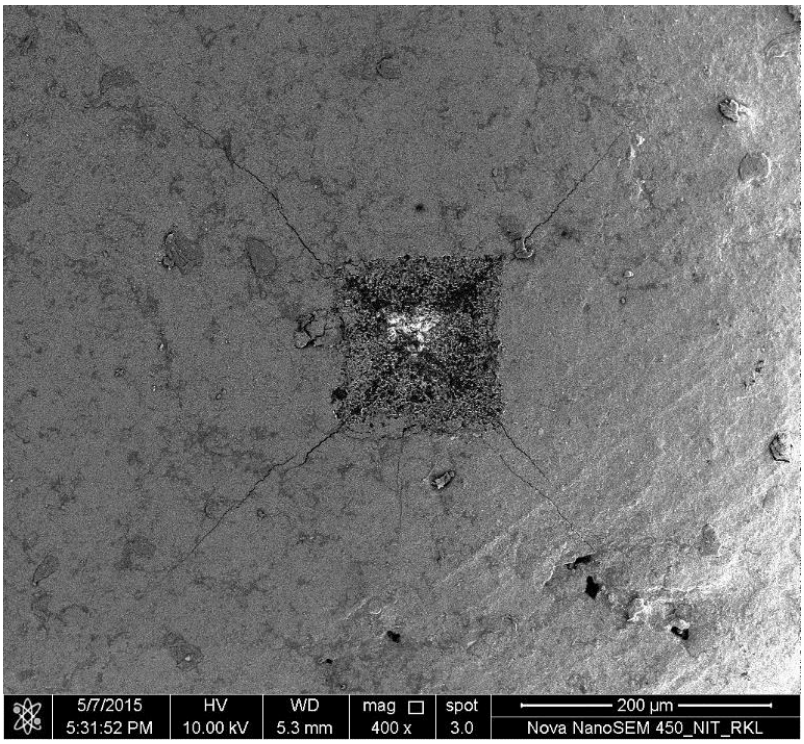

Fig. 10 INDENTATION IN ALUMINA SAMPLE

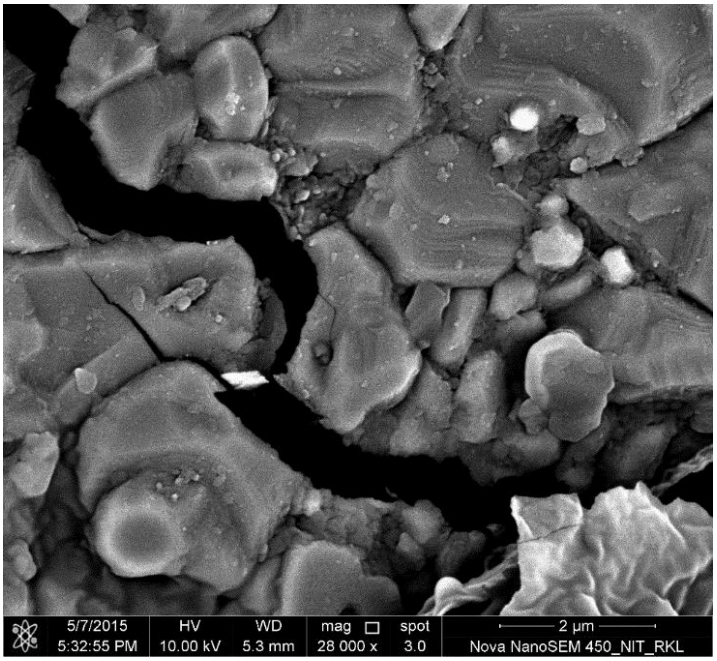

Fig. 11CRACK PROPAGATION ALONG THE GRAIN BOUNDARY

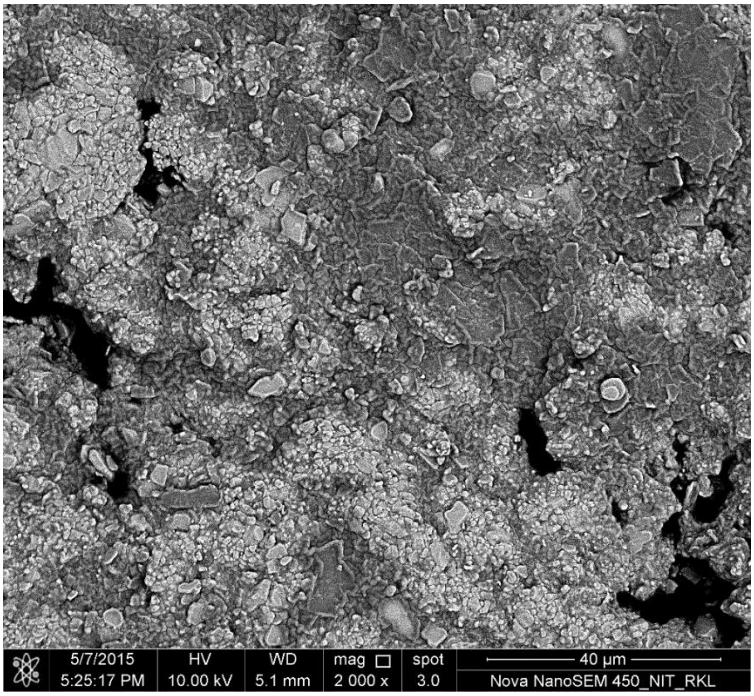

Fig. 12 POROUS MICROSTRUCTURE OF THE COMPOSITE

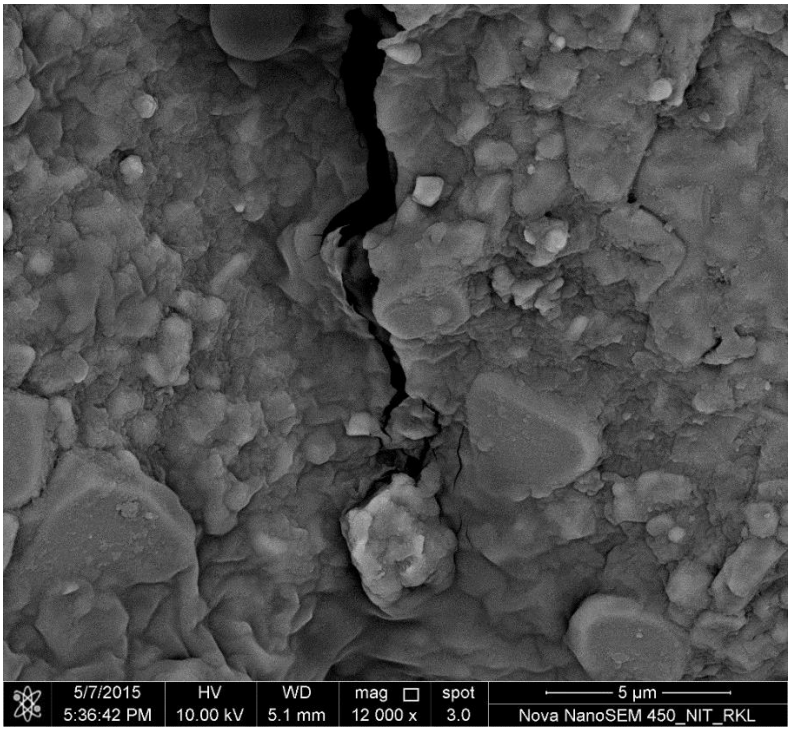

Fig. 13 STOPPING OF CRACK

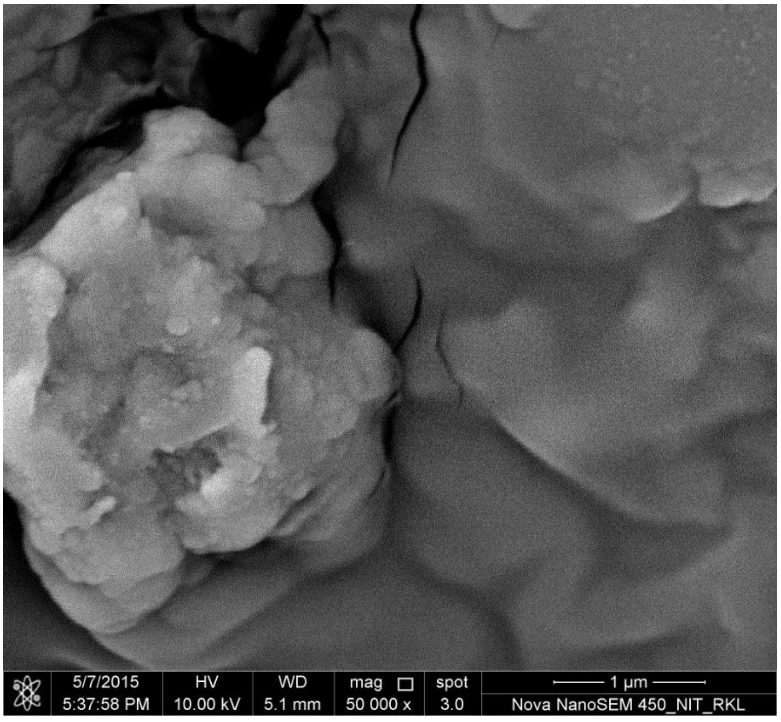

Fig. 14 CRACK BRANCHING

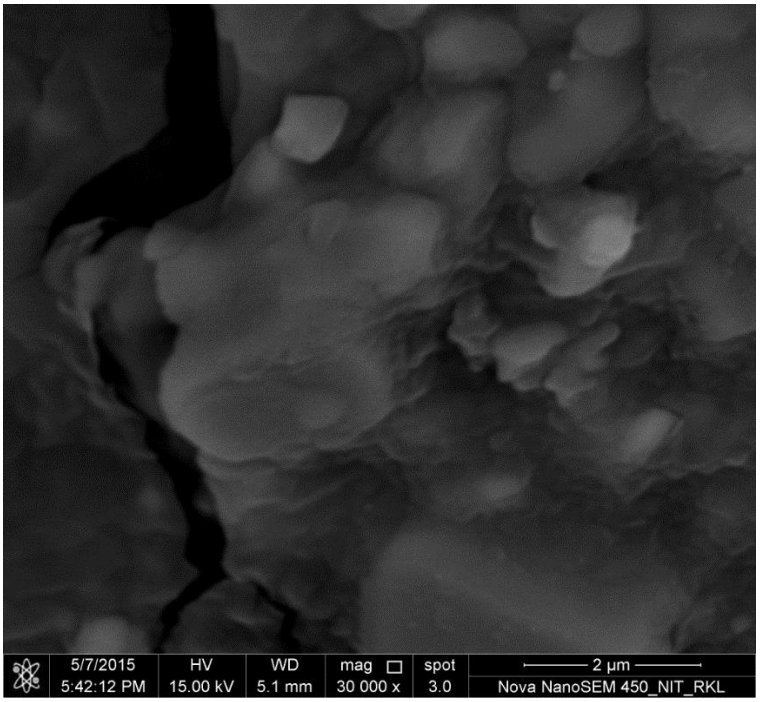

Fig. 15GRAIN CLUSTER OF $SrO.Al_2O_3$

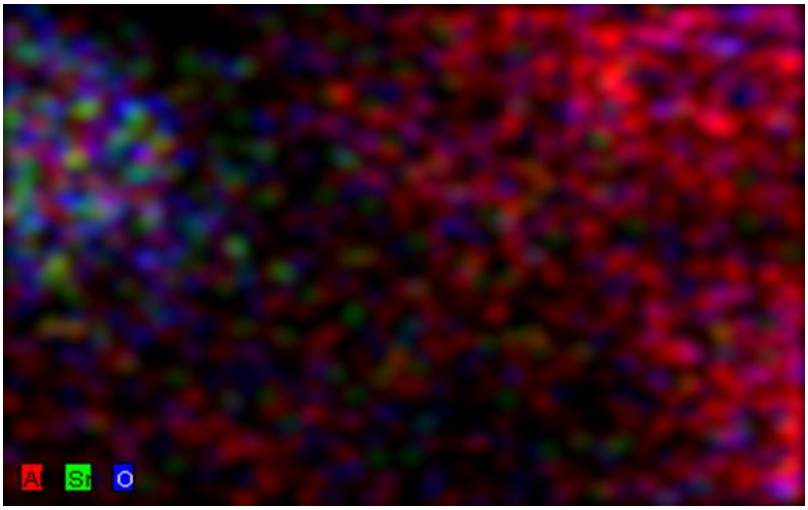

Fig. 16 EDAX PROFILE OF A BRIGHT GRAIN IN SINTERED ALUMINA-STRONTIUM ALUMINATE COMPOSITE

Fig 13 shows the crack path along the grain boundary of $SrO.Al_2O_3$. The crack appears to have stopped at the grain boundary of the bright phase $SrO.Al_2O_3$. Fig. 14 shows that the original crack is branching into many small cracks at the original strontium aluminate grain. Thus, crack branching is occurring in the vicinity of this lump that has the potential for increment. In fig. 15, it can be seen that the crack appears to stop or bend around this lump, thus restricting crack propagation. The grain cluster shows the presence of elongated and plate-like grains which is expected to restrict crack propagation. Thus, $SrO.Al_2O_3$ has the potential to increase toughness. Fig. 16 shows EDAX profile of the large grains. The profile shows that the grain contains Sr and Al and O, thus confirming the formation of $SrO.Al_2O_3$ phase.

6. CONCLUSIONS

CONCLUSIONS

Alumina -Strontium Aluminate ($SrO.Al_2O_3$) composites were prepared by mixing Strontium Nitrate with Calcined Alumina. The mixed powder was dried and calcined at 1100^oC. The calcined powder had corundum (Al_2O_3) and $SrO.Al_2O_3$ as the major phase. The calcined powder was compacted and sintered at 1600oC. The sintered samples were calcined characterized by AP, BD, Hardness, Biaxial Strength, Fracture Toughness and Microstructure. The DSC plot of the calcined powder showed an endothermic reaction is occurring at 600^oC, and the reaction involves nearly 7% weight loss. The broad exothermic peak at 1100^oCcorresponds to the phase formation reaction between SrO and Al_2O_3 leading to the formation of Strontium aluminate. From dilatometry, it is seen that strontium aluminate retards the densification of alumina. From XRD analysis, we found out that $SrO.Al_2O_3$ phase along with that of alumina is present in the composite.

From FESEM, it is found out that $SrO.Al_2O_3$ phase has been formed, and crack propagation is slowed near such grains. Thus, the composite has the potential to be tough ceramics.

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